

SOLUBILITY OF NANOFILLED VERSUS CONVENTIONAL COMPOSITES

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ABSTRACT

Solubility of resin-based composite materials is of great importance in restorative dentistry, since inorganic ions present as fillers within composites can leach into the surrounding environment resulting in breakdown of the restoration. The aim of this study was to evaluate and compare solubility values of three different nanofilled light-activated composites with other four conventional composite dental materials.

Seven commercial light-activated composite materials: Tetric Evo Ceram, Premise, Herculite, Z100, Z250, P60, SupremeXT. Ten disc specimens were prepared for each composite material using a stainless steel mold with 15 mm in inner diameter and 1 mm in thickness. The curing of each composite specimen was divided into five segments and each segment was photo-cures for 40 seconds. Water solubility of different materials was calculated by means of weighting the samples before and after water immersion (15 days) and desiccation. Data were analyzed by one-way ANOVA at 5% level of significance.

Tetric Evo Ceram composite showed the lowest solubility values, while Premise composite displayed the highest values. Solubility values of the tested composites did not show significant differences among them ($P > 0.05$).

All the composites being tested in this study exhibited solubility values within the acceptable limits and composite composition insignificantly influences its water solubility values.

Key Word: composite resin, composite solubility, Nanofilled.

INTRODUCTION

During the last few years, resin composites have been classified according to their filler particle size, as hybrid, microhybrid and microfilled. More recently, however, with the introduction of nanotechnology in dentistry¹⁸, a new class of resin composites, the so-called nanocomposites, is available to clinicians. However, there is a lack of data about the solubility phenomena of this new class of restorative materials. The hydrolytic degradation is a result of either the breaking of chemical bonds in the resin or softening through the plasticizing action of water.¹ When resin samples are immersed in water, some of the components, such as unreacted monomers or filler, dissolve and are leached out of the samples. This results in loss of weight and can

be measured as solubility or leaching.² Several factors contribute to the process of elution from dental composites: unreacted monomers, chemistry of the solvent and size and chemical composition of the elutable species.³ The release of these components may influence the initial dimensional change of composite,⁴ the clinical performance,⁵ the aesthetic aspect of the restorations⁶ and the biocompatibility of the material.³ Sorption and solubility are affecting composite restorations by two different mechanisms; the first is the uptake of water producing an increased weight and the second is the dissolution of materials (fillers or monomers) in water, leading to a weight reduction of the final conditioned samples.⁵

METHODOLOGY

Seven composite materials were selected for solubility evaluation in this study: Tetric Evo Ceram (nanofilled) (Ivoclar, Vivadent AG FL-9494 Schaan/Liechtenstein), Premise (nanofilled) (Kerr, orange, CA

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92867, USA), Herculite (Kerr, Orange CA 92867, USA), Z100 3M-ESPE, St. Paul, MN, USA), Z250 (3M-ESPE, St. Paul, MN, USA), P60 (3M-ESPE, St. Paul, MN, USA) and Supreme XT (nanofilled) (3M-ESPE, St. Paul, MN, USA) forming seven experimental groups ($n = 10$). The composition of the selected composites is summarized in Table 1. Each composite specimen disc was 15 ± 1 mm in diameter and 1 ± 0.1 mm thick and was prepared using a stainless steel mould (Figure 1). The material was prepared in accordance with the manufacturer's instructions, by filling the mold with the material using a plastic spatula to condense, and covering it with a polyester transparent film which was placed over the mould and finally covered by a glass slide. The photo-curing of each composite specimen was divided into five sections overlapping each other and each section was photo-cured with the bluephase C5 (LED) (Ivoclar, Vivadent AG, FL-9494 Schaan/Liechtenstein, Austria) light curing unit for 40s. The specimens were removed from the mould and any flash if present, was removed. The specimens were transferred to an air oven (memmert, GmbH, D-91126 Schwabach, Made in Germany) and dried for 2 hours at 37°C . Then the specimens were transferred to the desiccators containing silica gel, freshly dried for 2 hours at 20°C . The specimens were weighed using an analytical balance (Precisa, TYP 205A, made in Switzerland) to an accuracy of ± 0.1 mg. This cycle was repeated until a

constant mass (m^0) was obtained. The specimens were immersed in distilled water and maintained at 37°C for 15 days. After that time, the specimens were removed, washed with water, surface water blotted away until free from visible moisture, and waved in the air for 15 seconds, then the specimens were placed in the desiccator using the same cycle as described above but the temperature was 58°C to obtain (m_1). This cycle was repeated until constant mass was obtained. These steps were carried out to evaluate solubility (S) according to Oysaed & Ruyter (7) formula: $S = \frac{m^0 - m_1}{V}$, where m^0 is the sample weight before water immersion, m_1 is the sample weight after immersion and desiccation. V is the volume of the specimen in cubic millimeters. Solubility data was analyzed by one-way ANOVA at 5% level of significance.

RESULTS

Table 2 summarizes solubility means and standard deviations (in parenthesis) of Tetric Evo Ceram, Premise, Herculite, Z100, Z250, P60 and Supreme XT composites in $\mu\text{g}/\text{mm}^3$. Solubility evaluation indicated that, Premise light activated composite exhibited the highest mean value, while Tetric Evo Ceram composite exhibited the lowest mean value (Figure 2). Further statistical analysis of data by using one-way analysis of variance (ANOVA) revealed that, there was statistically

TABLE 1: COMPOSITION OF THE SELECTED COMPOSITES IN THIS STUDY

Compo-site	Organic/inorganic Matrix	Inorganic Filler	Filler size	% in Vol-ume (Filler)
Tetric Evo-Ceram	Dimethacrylates (17-18% weight)	barium glass, ytterbium trifluoride, mixed oxide and prepolymer	0.04-3 μm 0.55 APS	*61-60
Premise	The ethoxylated Bis-GMA	Non-agglomerated silica nanoparticles, prepolymerized filler, 0.4 micron barium glass	0.02 μm APS	69*
Herculite	Bis-GMA and TEGDMA	Barium glass and silicon dioxide	0.6 μm	59%*
Z100	Bis-GMA and TEGDMA	Zirconia/Silica	0.01-3.5 μm	66*
Z250	Bis-GMA, UEDMA and Bis-EMA	Zirconia/Silica	0.19 – 3.3 μm	60*
P60	Bis-GMA, UEDMA and Bis-EMA	Zirconia/Silica	0.01-3.5 μm	61*
Supreme (XT)	BIS-GMA, BISEMA, UDMA and TEGDMA	Zirconia/Silica	(clusters of 0.6 to 1.4 μm particules of 5 to 20 nm)	57.7*

* Manufacturer's information

APS: Average particle size

TABLE 2: SOLUBILITY MEANS AND STANDARD DEVIATIONS OF THE TESTED COMPOSITES IN $\mu\text{MG}/\text{MM}^3$

Composite	Solubility
Tetric Ceram	2.639 (1.331)
Premise	4.359 (1.534)
Herculite	4.020 (0.903)
Z 100	2.901 (1.787)
Z 250	2.774 (2.109)
P 60	2.718 (1.705)
Supreme XT	4.020 (1.497)

TABLE 3: ONE-WAY ANALYSIS OF VARIANCE (ANOVA) OF THE SOLUBILITY TEST

Source	DF	SS	MS	F	P
Factor	6	33.54	5.59	2.21	0.054
Error	63	159.57	2.53		
Total	69	193.11			

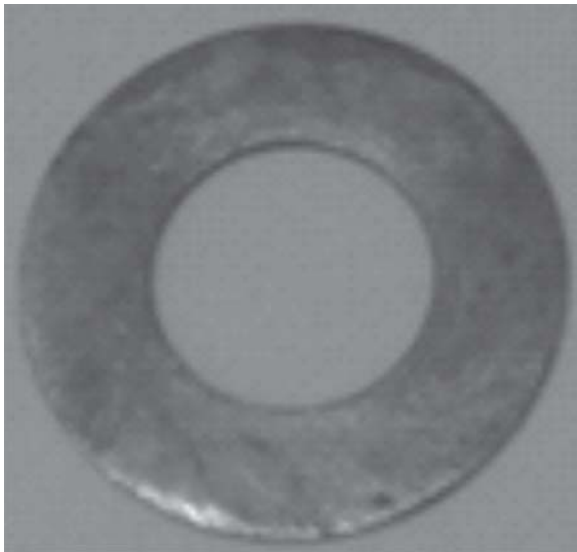


Fig 1: The composite stainless steel mould used in this study.

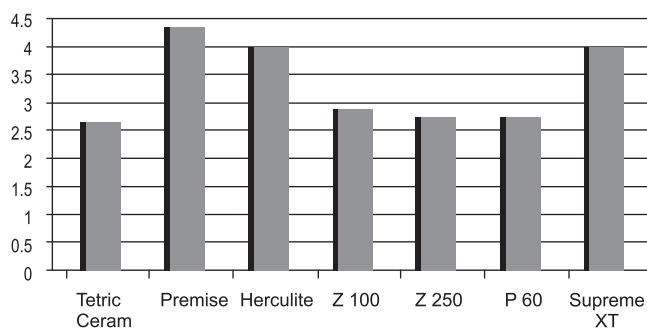


Fig 2: Mean solubility values for the composite groups in $\mu\text{g}/\text{mm}^3$.

insignificant difference ($P \geq 0.05$) in solubility values between the seven composite groups being tested as shown in Table 3.

DISCUSSION

Solubility of resin-based composite materials is of concern, since some composite inorganic filler ingredients can leach into the surrounding environment resulting in breakdown of the restoration. Higher solubility values can manifest as reduced wear and abrasion resistance of resin-based composites, as well as color instability. ADA Specification No. 27⁸ requires that “the solubility of all materials shall be less than or equal to $7.5 \mu\text{g}/\text{mm}^3$ within a seven day period of water storage” Resin composites indicated as restorative materials must also comply with ISO 4049 for a maximum value of $7.5 \mu\text{g}/\text{mm}^3$ for water solubility within a seven day period of water storage.⁹ Solubility values obtained in this study are remarkably lower than ADA and ISO guidelines, even for a 15-day storage time which is double than the recommended time.

Several factors, such as composite organic continues resin phase,¹⁰ composite inorganic dispersed filler phase,¹¹ and the degree of conversion reached after the polymerization reaction¹⁰ can influence the solubility and sorption behavior of resin composites. In the present study, insignificant differences were detected between the composite groups (Table 3). Results were probably related to the composition of the tested composites. The composites used in this study had a great similarity in the filler particle content by volume except for Premise light activated composite (nanofilled) which exhibited a relatively high solubility values and this could be related to its highest filler loading by volume (69%) and the presence of non-agglomerated silica nano-particles and prepolymerized filler (Table 1) and this coincide with the findings of Da Silva et al (2008)¹² who found that, nanofilled composites may exhibit higher solubility values in the oral environment than hybrid ones if it was inadequately polymerized. The other possible cause behind the highest (but insignificant) mean values in water solubility associated with Premise light activated composite could be due to the resin matrix composition since Premise is the only tested composite that contains ethoxylated Bis-GMA resin matrix which can be considered a weak resin matrix in providing the composite adequate resistance against solubility or due to the incorporation of prepolymerized filler in its structure that might interfere with adequate curing of this composite.¹⁰ In general we can say that all the

composite in this study were adequately polymerized and this cause might explain also (beside the comparable structure) the comparable solubility values in distilled water (Table 2) (insignificant differences) that were detected between the composite groups and their general lower values than ADA and ISO recommended limits and this might be attributed to the fact that, The solubility values for most of the composites were significantly higher in artificial saliva than in distilled water.¹³

CONCLUSION

All the composites being tested in this study exhibited solubility values within the acceptable limits. Nanofilled composites being tested in this study exhibited comparable solubility values with other conventional hybrid composites.

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